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ELECTRON-BEAM-INDUCED DAMAGE IN PAINT SAMPLES.(U)
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Electron-beam-induced damage was investigated in paints that are being developed as protective coatings for missiles. The electron-beam testing of the paints had four objectives: (1) to determine, by establishing the damage threshold and mode, if low-level stress would cause pop-off, (2) to determine the impulse as a function of fluence, (3) to determine the mass loss as a function of fluence, and (4) to determine the Grüneisen parameter for

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the paints. The tests showed that the damage mode was not low-stress-level pop-off, but spall at a fluence of 30 to 40 cal/cm^2 incident on the filters. The tests showed also that the impulse as a function of fluence is steplike. At 25 to 30 cal/cm^2 fluence, the impulse is too small to be measured, but at 40 cal/cm^2 it increases to about 1 kilotap and remains essentially at that level at higher fluences. By using stress-time measurements, the Grüneisen parameter was calculated to be 0.37 if crushup and plastic work are neglected. This value, therefore, represents a lower bound on the parameter. Both impulse and the mass-loss data showed large variations at each fluence level. Further analysis indicated that these data variations were caused by experimental problems resulting primarily from the use of the filters and the wide variation in the response of the different paint samples.

CONTENTS

FIGURES

1. INTRODUCTION

Electron-beam-induced damage was investigated in paints that are being developed as protective coatings for missiles by the Defense Nuclear Agency. The materials development and underground testing were performed by the McDonnell Douglas Astronautics Corporation (MDAC). The electron-beam tests to simulate some of the conditions and effects in underground tests (UGT) were performed by the Harry Diamond Laboratories (HDL) at the Naval Research Laboratories' Gamble I Facility.

The primary objectives of this study were to investigate the possibility of low-stress-level pop-off--that is, the removal of the paint when a low stress level destroys the bond at the paint-substructure interface--and to determine if pop-off was a potentially serious problem. Additional experimental objectives were to obtain the impulse and the mass loss as a function of fluence, to measure the stresses produced in the paints, and to determine the Grüneisen parameter of the paint. Then data from the computer calculations could be compared with UGT results to determine the accuracy with which the parameters of the materials are known and to determine if the electron beams adequately simulated the UGT conditions and effects.

2. PROCEDURES

Since a major objective was to simulate x-ray effects, it was desirable to simulate an x-ray energy deposition profile. Initially, the profile was selected by the energy spectrum of the electron beam, and then it was further tailored by filtering the beam with a material of the correct density and thickness. For these experiments, profiles were calculated with the computer code ZEBRA[1] for several electron-beam spectra and filters. Although the best profile was obtained for a 10-mil (2.54×10^{-2}-cm) titanium filter and the 550-keV electron spectrum from the Gamble I Facility, titanium was observed to adversely affect the operation of Gamble I, and the filter material was later changed to carbon, which produced a satisfactory profile.

Two types of samples were fabricated by MDAC. For the impulse and damage threshold measurements, the samples consisted of approximately 20 mils (5.08×10^{-2} cm) of paint on a carbon-phenolic substrate ($2.54 \times 2.54 \times 0.76$ cm). This configuration simulated the exterior of a missile system. The samples for the stress, impulse, and mass loss

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[1] *Lawrence D. Buxton, The Electron Transport Code ZEBRA 1, Harry Diamond Laboratories TR-1536 (June 1971).*

measurements consisted of approximately 20 mils (5.08×10^{-2} cm) of paint on a fused-silica disk (0.203 cm thick, 3.175 cm in diameter). The fused-silica substrates were thin disks that were polished to optical flatness so that they could be used for either laser interferometer or quartz gage measurements. The types of paint that were tested, for both the carbon-phenolic and fused-silica substrates, were MAP-MNCr-6, LTB-2AK2, and LTB-2AK4.

Initially, the carbon substrate samples were mounted on the ballistic pendulum bob so that both the damage threshold and impulse could be determined with a minimum number of shots (fig. 1). However, for low fluences (less than 20 cal/cm^2), a reproducible electron beam could be obtained only if the samples were placed very close to the anode. With this experimental arrangement, reproducible, uniform beams were produced, but the pendulum could not be used with this configuration. When it was found that the damage threshold was above the 20-cal/cm^2 fluence level, the impulse measurements were made simultaneously. After the damage threshold was determined, the guide cones and the samples on fused silica were used so that both impulse and stress could be measured on each shot (fig. 2).

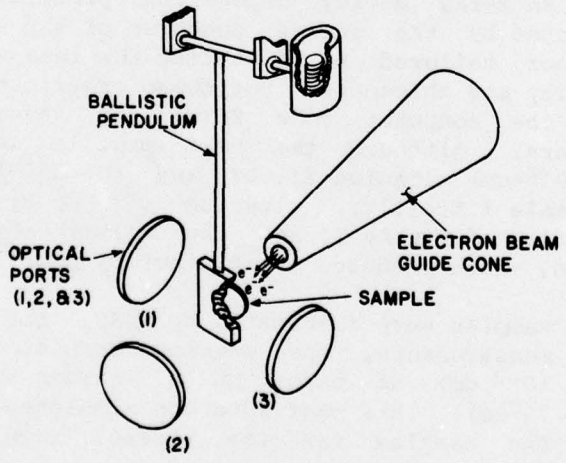

Figure 1. Experimental setup to measure impulse with ballistic pendulum.

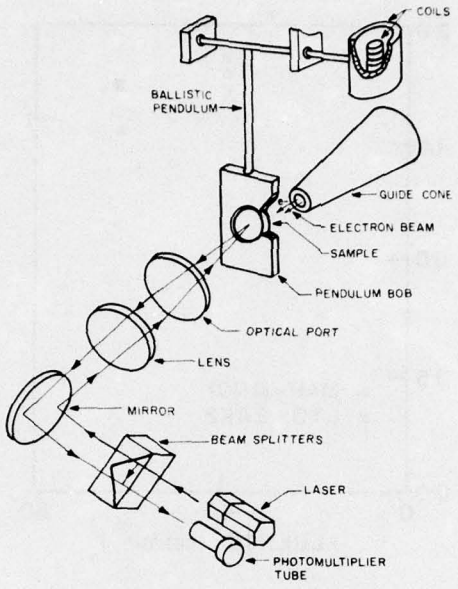

Figure 2. Experimental setup to measure impulse and rear-surface displacement simultaneously with ballistic pendulum and laser interferometer.

3. RESULTS

To determine if pop-off, a low-level phenomenon, would be a problem, the damage threshold was investigated. At a fluence level of 25 cal/cm^2, no damage was evident and no appreciable mass loss was observed. At a fluence level of 38 to 40 cal/cm^2, all the samples were damaged and had specific mass losses that ranged from 2.85×10^{-2} to 4.15×10^{-2} gm/cm^2. The damage threshold was found to be between 30- and 40-cal/cm^2 incident fluence on the 25-mil (6.35×10^{-2}-cm) carbon filters. Attempts to establish the damage threshold more precisely were unsuccessful due to sample variation and fluence fluctuation. However, since pop-off would have occurred at an interface and below a fluence level of 30 cal/cm^2, and since all of the damage observed within the range of the damage threshold was internal spall and not bond separation, pop-off is not considered a failure mode.

After the damage threshold and failure mode were established, the impulse and mass loss as a function of fluence were studied. Figure 3 shows the measured impulse and the specific mass loss as a function of fluence incident on a 25-mil (6.35×10^{-2}-cm) filter, for paint samples

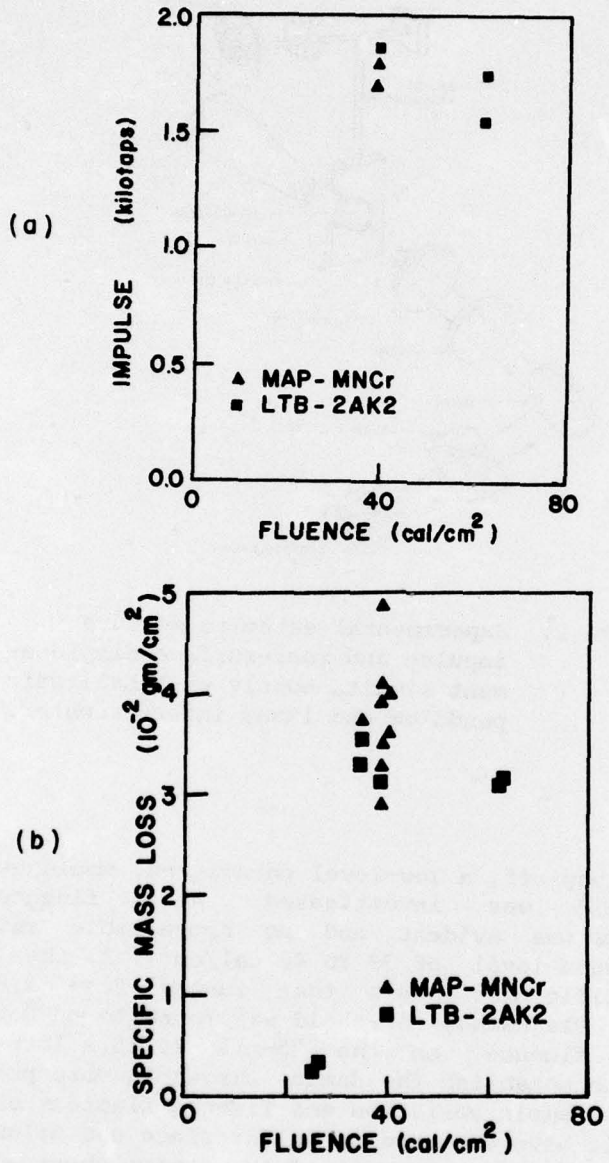

Figure 3. Carbon-phenolic substrates; fluence incident on 25-mil (6.35×10^{-2}-cm) carbon filter: (a) impulse versus fluence and (b) specific mass loss versus fluence.

mounted on carbon-phenolic substrates. The predicted impulse as a function of fluence as calculated by MDAC was an order of magnitude lower than the impulse measured in the test. However, the quartz gage measurements of stress agreed well with the calculated stress. This result implied that the discrepancy in the impulse could have been caused by an improper calculation or an experimental problem with the stress gage measurement, the fluence variation, the anode debris, or a spurious impulse generated by something other than the sample. A combined stress and impulse measurement was the best method to resolve the discrepancy. To measure the impulse, the paint samples with fused-silica substrates were mounted on a ballistic pendulum so that a laser interferometer could be used to record the response of the material to determine the stress (fig. 2). Figure 4 shows the impulse and specific mass loss as functions of fluence incident on a 30-mil (7.62×10^{-2}-cm) filter.

The increase in filter thickness produced a change in the depth-dose profiles (fig. 5), which must be considered when interpreting the results. Consequently, the impulse or mass loss obtained with the two different filters cannot be compared on the basis of fluence incident on the filter. Although results could be compared on the basis of front-surface dose for the paint, that approach does not resolve the difficulty, because there is a linear relationship between the front-surface dose and the incident fluence on the filters. (The front-surface dose in calories per gram for the 25-mil (6.35×10^{-2}-cm) filter is approximately 5 times the incident fluence, and for the 30-mil (7.62×10^{-2}-cm) filter, it is approximately 3.5 times the incident fluence.) Comparison on the basis of the total energy deposited in the paint (fig. 6) is probably more appropriate.

When the impulse was plotted against the energy deposited in the paint, the impulse recorded for the samples with a carbon-phenolic substrate was significantly higher than that recorded for those with fused-silica substrates. This result could be either due to significant differences between the stress wave propagation across the interface for the two cases or due to the inelastic work being done on the fused silica. In either case, this result supports the use of the carbon-phenolic substrates to simulate actual conditions.

Three characteristics of the impulse can be seen in figure 3(b). The first is the fairly wide variation in the data at each fluence level. The second is the steplike increase in the impulse; i.e., at 25 cal/cm^2, no impulse was recorded, but at 40 cal/cm^2, approximately 1 kilotap was measured. The third is the apparent insignificant change in impulse between 40 and 60 cal/cm^2. This last characteristic is difficult to establish with certainty because of the wide variation in impulse at each fluence level. This variation could be the result of

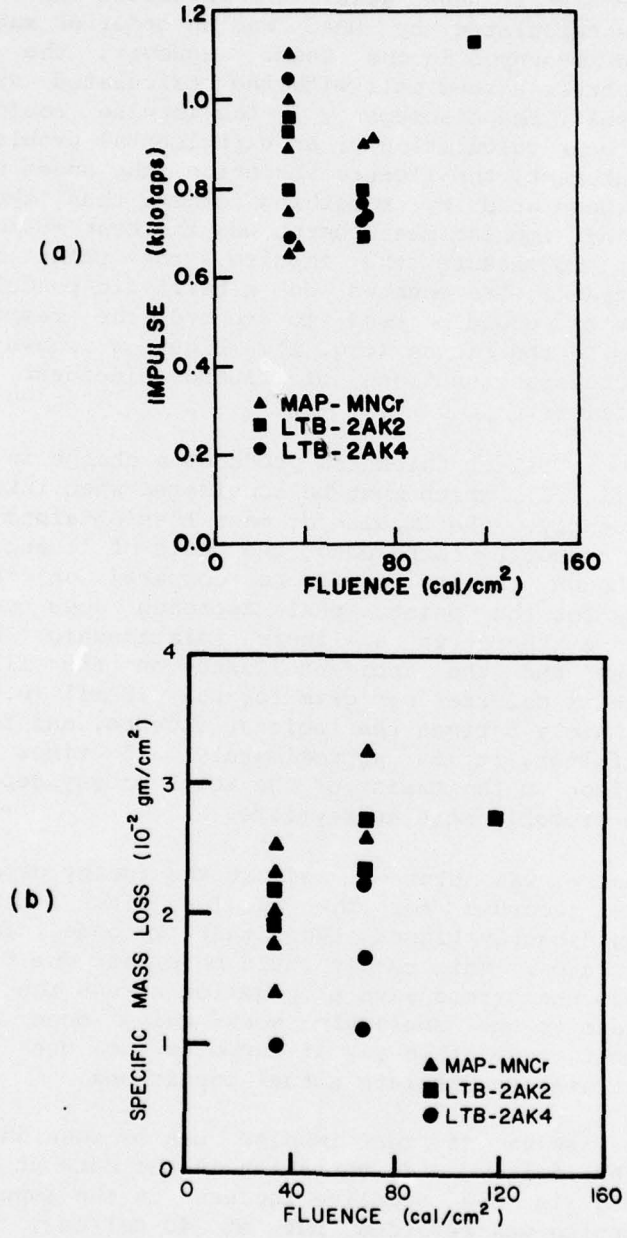

Figure 4. Fused-silica substrate; fluence incident on 30-mil (7.62×10^{-2}-cm) carbon filter: (a) impulse versus fluence and (b) specific mass loss versus fluence.

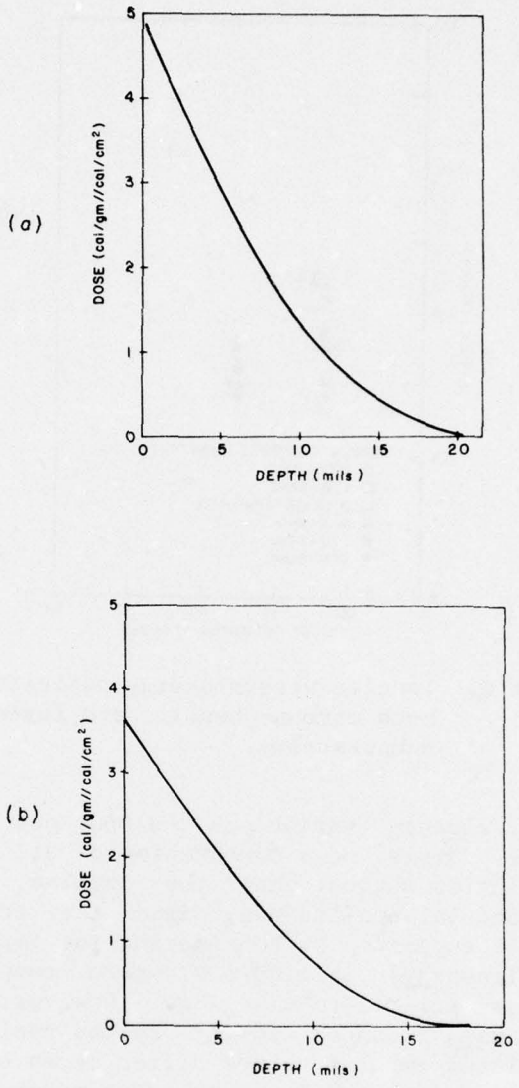

Figure 5. Depth-dose profile in paint: (a) 25-mil (6.35×10^{-2}-cm) and (b) 30-mil (7.62×10^{-2}-cm) carbon filter.

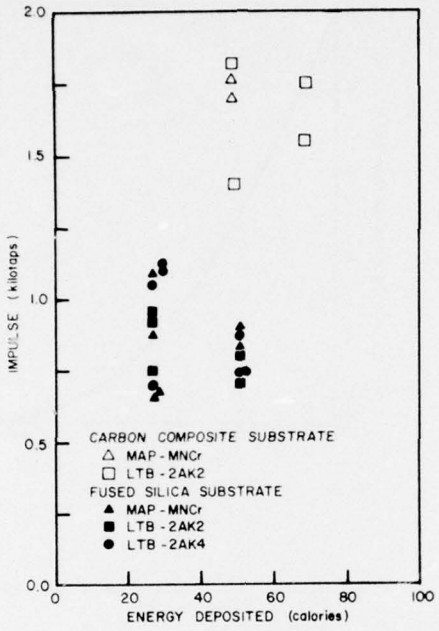

Figure 6. Impulse versus energy deposited for both carbon-phenolic and fused-silica substrates.

material variations, fluence variations, a spurious impulse from something other than the sample, or a combination of all three. The second and third characteristics suggest that the impulse resulted primarily because the paint material spalled away from the front surface. This conclusion is further supported by considering the impulse as a function of mass loss, as illustrated in figure 7, where comparison shows the impulse to be directly related to mass loss. However, if it is assumed that the impulse was produced solely by the spalled material, the measured and calculated impulse values differ by an order of magnitude, but the stress obtained from the laser interferometer measurements agree reasonably well with the measured values. These two considerations indicated that the only experimental problems that could account for the discrepancy were anode debris and a spurious impulse.

To investigate these two possibilities, shots were made with a carbon sample mounted on the pendulum, with and without the carbon filters in front of the sample with the sample either touching or not touching the guide cone. The impulse that was recorded from all of these shots indicated the following: (1) the samples can receive a small amount of impulse from the guide cone; therefore, a small gap between the sample

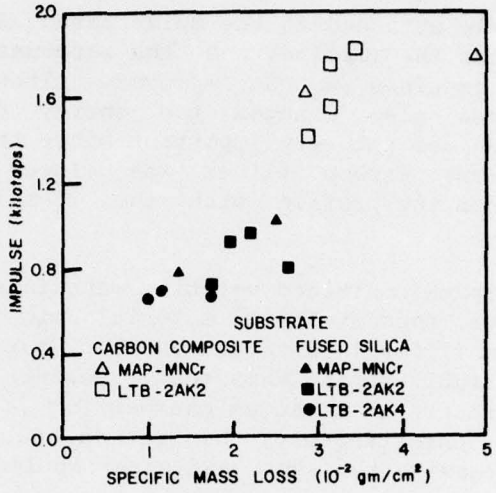

Figure 7. Impulse versus mass loss for both carbon-phenolic and fused-silica substrates.

and the cone is necessary for good measurements; (2) the anode debris at the fluence levels for these tests did not cause the discrepancy; and (3) no measurable impulse was created in the carbon samples without the filter, but if carbon filters were in front of the carbon sample, some impulse was created. This somewhat ambiguous result was probably due to volatile materials in the carbon filters that are not present in the sample. Perhaps the same volatile materials were present in the carbon sample originally, but after a number of shots they were vaporized and driven out of the sample. A similar procedure might have been used to purify the filters, but unfortunately most of the filters could not survive the first shot. Another possible reason for increased impulse when the filters were in place could be the tamping effect that they would have on vapors being ejected from the samples.

To eliminate the spurious impulse created by the filters and to decrease the vulnerability of the filters, their thickness was increased from 25 to 30 mils (6.35 to 7.62 $\times 10^{-2}$ cm). These filters were composed of two layers: (1) a 20-mil (5.08×10^{-2}-cm) layer and then a 5-mil (1.27×10^{-2}-cm) layer for the 25-mil (6.35×10^{-2}-cm) filter and a 20-mil (5.08×10^{-2}-cm) layer, and then a 10-mil (2.54×10^{-2}-cm) layer for the 30-mil (7.62×10^{-2}-cm) filter. For both filters, the thin layer faced the electron beam and acted as a filter for the thicker layer. This configuration did improve the survivability of the thicker layer, which could reduce the impulse delivered to the samples, since

the filters were firmly attached to the guide cone. But this change did not significantly alter the impulse, and the agreement between the calculated and measured impulses was not improved. Although the change in the filter thickness also changed the energy deposition profile (fig. 5), this change was not very important since the profile with the 30-mil (7.62×10^{-2}-cm) carbon filter was closer to the titanium filtered profile than the profile with the 25-mil (6.35×10^{-2}-cm) carbon filter.

If the carbon filters contained volatile materials that created this survivability problem, these volatile materials might have been removed by heating the carbon filter in a vacuum chamber, but the equipment to do so was not available during this test. However, the filters were placed at room temperature in a vacuum chamber for several hours to try to remove volatiles. Unfortunately, this method was not successful: the filters did not survive the shot, and some impulse was transmitted to the sample.

The large, spurious impulse could have been created by tamping the vapors from either the filters or the samples, or it could have been created in the filters and mechanically transmitted to the sample. To resolve this issue, a number of shots without the filters would be required. Two such shots with a sample on the pendulum were attempted, but the substrate was seriously shattered, so it is doubtful that the total impulse was measured. Thus, the source of the spurious impulse is not completely explained.

The Grüneisen parameter was determined by using a displacement interferometer.[2] The samples for these measurements consisted of 20 mils (5.08×10^{-2} cm) of paint deposited on polished-quartz substrates with a reflective coating on the free surface. On most shots, useful data were unobtainable because either the substrate shattered or the photomultiplier trace was unclear. However, at the 34-cal/cm^2 fluence level, three shots produced useful data. The measured displacement was between 4.9×10^{-5} and 5.8×10^{-5} cm, so that the average peak displacement was 5.2×10^{-5} cm. The previous analysis[2] was expanded to include the effect of the interface between the paint and the substrate by using the method developed by F. B. McLean[3] of HDL. In addition, relaxation effects during the energy pulse were taken into account. The calculated Grüneisen parameter is 0.37. This value must be considered a lower bound, because the crushing

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[2] *R. B. Oswald, Jr., F. B. McLean, D. R. Schallhorn, and L. D. Buxton, One-Dimensional Thermoelastic Response of Solids to Pulsed Energy Deposition, J. Appl. Phys., 42, No. 9 (August 1971), 3463.*

[3] *F. B. McLean, A Note on Front Surface Thermoelastic Relaxation, Harry Diamond Laboratories TM-76-24 (August 1976).*

of the porous paint material and the plastic work caused by shear stresses have been neglected. These effects were not included because the parameters required are not yet known. If these effects had been included in this study, the measured Grüneisen parameter would have been larger.

4. CONCLUSIONS

Because there was significant scatter in the experimental data, the impulse results could not be interpreted completely. The stress and mass-loss measurements were unaffected by the problem created by the filters, and the data obtained in these two areas, especially the mass loss, suggest that the main causes of the scatter in the experimental data were the exposure variation and material variation. The material variation probably was the more important of the two, if the exposure variation was similar to that observed in the past.[4]

Even when the scatter is taken into consideration, the data were sufficient to make several conclusions concerning effects of electron beams on the paints that were tested: (1) low stress did not cause pop-off, (2) the surface dose damage threshold is between 150 and 200 cal/gm for the beams used, (3) the primary damage mode at or near threshold is spall, (4) the Grüneisen parameter is 0.37 when crushup and plastic work are neglected, and (5) the impulse is much higher than would be predicted by tensile stress, which suggests that extraneous materials may be vaporized, causing the impulse to be an order of magnitude greater than the predicted impulse. (Aerospace and MDAC will investigate this area further.)

To define impulse as a function of fluence accurately, some additional electron-beam tests would have to be performed. These tests would have to be designed differently from the tests described in this report in order to solve the problems created by the filters. The filters would have to be either heated in a vacuum chamber to eliminate volatiles or omitted. At this time, omission of the filters appears to be the better alternative, since the filters would always create the problem of tamping any vapors emitted by the paint samples.

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[4] *D. R. Schallhorn, G. L. Skillington, R. B. Oswald, Jr., and T. R. Oldham, Some Characteristics of the Gamble I Electron Beam, Harry Diamond Laboratories TR-1677 (December 1974).*

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